

1001502391

SUPERVISOR'S MEETING MINUTES

1957

Sample	Original (Analysis)	Check	Diff.
Series 2 - 3	.53%	.45	.08%
12	.49	.47	.02
21	.43	.48	.05
Series 4 - 3	.62	.59	.03
8	.58	.58	.00
12	.49	.48	.01
21	.58	.55	.03
Average Diff.			.03%

4-003P Material Balance in the BL Process (Pleasants, Wiley, Haynes)

During the early part of August, 6 samples were taken from each of the 3 cooks at the BL plant. Chemical analysis was run on these samples. It was obvious that I did not get enough samples to have a representation of the batch.

Samples were taken from another batch following a complete sampling plan. 28 samples were taken and these consolidated to 20. Those analyses appear to follow a pattern. Chemical analysis from another batch are now in the process of being run. It is a fair amount of trouble to do the sampling. It is believed that if tests are taken from 5 batches a fair picture of material loss can be made.

4-004 Smoking Machines (Sowell, Cosby)

Both the 8 and 20 port smoking machines were operated on routine analysis during this period and the following smoke analyses were completed: 136 tar and 169 nicotine.

Special tests have been made for the Quality Control Division.

/hjl

cc: Dr. R. N. DuPuis
Mr. C. C. Cosby, Jr.
Mr. L. L. Long
Mr. J. D. Hind
Dr. S. M. Cantor

1001502395

4-002 Comparison of Brands (Sowell, Cosby)

Moisture determination and pack inspection has been completed on cigarettes from the Chico pickup.

Tests are being conducted on special cigarettes from Mr. E. J. Gray.

4-003A Leaf Analysis (Pleasants, Wiley)

224	Nicotine	62	Petroleum Ether Extract
84	Ash	164	Total Nitrogen
96	T.V.B.	188	Sugar
184	Moisture		

Total 1002

4-003D Miscellaneous Laboratory Tests (Stewart, Cosby)

10-20 Packaging Materials: The conditioning of the packaged cigarettes in containers from Milprint is nearly complete.

Final evaluation will be made at the end of the conditioning period.

4-003D Miscellaneous Laboratory Tests (Estes, Hellams, Butler)

A. Determination of Total Organic Acids in Cigarette Smoke: Eighteen total acid titrations by the differential titration method were made on a composite smoke sample representing fifty PM regular cigarettes in 500 ml of ethanol-toluene. The results of this test gave a value of 0.0291 milliequivalents of acid per cigarette with a two sigma value of 0.0024 milligrams.

B. Nitrogen in Tobacco by Breakdown: Another USDA tobacco sample is being carried through the nitrogen by breakdown method as described by Dr. Frankenburg. A complete analysis of the nitrogen breakdown is available and this analysis will be used to compare our results with those obtained by Dr. Frankenburg.

C. Carbonyl content of Smoke by 2,4-DNP's: The investigational outline previously referred to is nearing completion. This should complete the experimental work on this project.

4-003D Miscellaneous Laboratory Tests (Pleasants, Wiley, Haynes)

Sulfur: A total of 73 analyses were made for sulfur during this period. Every 5th sample is run in duplicate. A statistical check was made on USDA samples. Some of these are bright and some burley tobacco. The precision of the method appears to be good. The results on seven samples are listed below:

1001502394

... from water cooked stem pulp. The physical properties compare favorably with present BL production with exception of breakage and sheet weight.

ATBL was prepared from tobacco fines, without cooking. (The tobacco fines used are those presently used in BL.) The material looks similar to BL. Tensile strength is fair.

-7-

2-006A Shredded Stems (Super, Long, Michels)

Arrangements have been made for the Herty Foundation to shred 500 lbs. of stems using the same conditions as were used in July. Two hundred pounds of these stems will be sent to Sprout-Waldron for screening tests during the second week in September. The purpose of the screening tests is to determine the design of the screen that would be needed to produce material in the size range of -10 +30 mesh in a commercial process.

The other 300 lbs. of shredded stems will be used for other purposes as needed.

It is planned to make firmness measurements on cigarettes made with filler containing 0, 10, 20, and 50% shredded stems. If the filling power has been increased by incorporating shredded stems in the blend, it may be possible to make a cigarette with less tobacco; hence, less nicotine and tars in the smoke.

2-006B-1 Analysis of Formaldehyde in Stem Pulp (Clarke, Moore, Butler)

A Beer's law graph has been prepared on formaldehyde for the purpose of analyzing tobacco stem pulp for formaldehyde by the chromotropic acid method. Results from the formaldehyde analysis of pulp indicate that the method can be used. Statistical analysis will be run for confirmation.

2-006B Miscellaneous (Clarke, Moore, Butler)

ATBL from Water Cooked Stem Pulp: ATBL films were made from water cooked stem pulp. Molins dust, PM flavors and glycerine were added to the pulp. The films were evaluated by the Physical and Analytical Sections.

Physical Tests

Determinations	ATBL	BL Average	Range
Dust Loss, %	8.1	7.0	4.3 - 11.6
Breakage, %	38.5	7.3	2.7 - 18.0
Moisture, %	12.7	12.9	12.4 - 13.7
Dry Sheet Wt., % (gm./sq.ft.)	17.2	7.8	7.3 - 11.3
Filling Power, % (cc/10 gm.)	42.0	8.0	34 - 40

Tensile Strength = 1.164 kg/in.

Tensile coefficient = .06

BL tensile coefficient range = .06 - .1

1001502390

when 25 mg. was laboratory cased on uncased PM filler. Blanks for uncased tobaccos were: MF - 3.0 mg/g; PM - 2.7 mg/g; burley - 1.3 mg/g; BL - 2.0 mg/g. The value for a Benson & Hedges cigarette was 3.4 mg/g. Inositol does not interfere with the determination of glycerol.

-3-

to be 1.75 ms. and the sensitivity at this point was 1×10^{-10} grams/mv. This sensitivity is far greater than the sensitivity of the detector presently being used by the gas chromatography group. This new gauge will be turned over to the gas chromatography group for evaluation.

1-003F Instrumental Research and Service - Infrared Spectroscopy (Hill, Vilcins, Resnik, Harrow)

Various packaging materials obtained from Milprint, Inc. were examined as films using the infrared spectrophotometer. These materials are being analyzed as reference standards.

The neutral fraction of the petroleum ether extract was separated chromatographically by the Organic Section. Infrared analysis of these separated fractions showed the presence of solanesol, waxes, and glycerides.

Work is continuing on the quantitative analysis of smoke by infrared spectroscopy.

1-003G Service and Research - Ultraviolet Spectroscopy (Kuhn, R. Resnik, F. Resnik, Harrow)

Numerous service samples were analyzed for Mr. Edmonds (nicotine determinations), Mrs. Estes (mono- and di-carbonyl determinations), Dr. Seligman (unique 2,4-DNP derivatives), Mr. Murrill (gas chromatographic fractions), Mr. Carpenter (petroleum ether fractions) and Mr. Martin (formaldehyde-chromotropic acid complex) during this period. The results of these analyses will be reported under each individuals' respective projects.

Several vapor state spectra, benzene, toluene, pyridine and isoprene, were obtained as reference standards. These gas phase spectra are a basis for greater utilization of the ultraviolet spectrophotometer in the analysis of gas chromatographic fractions.

1-009 Physical Studies of Tobacco and Smoke (Holmes)

Studies on WIN, WIN-V, and NIW cigarettes were completed. The maximum temperatures were as follows:

Cigarette	Temperature	
	Center	1 mm from edge
WIN	868°C	843°C
WIN-V	894	862
NIW	881	963

All these values are the same within the limits of experimental error.

1001502386

on pulp and paper, will be the speaker and will talk on pulping processes. He has received the TAPPI award, one of the highest awards in the paper industry.

Mr. O'Keeffe would like to have suggestions for the October seminar submitted to him.

-2-

1-002H Miscellaneous - Formaldehyde-DNP (Uram)

Formaldehyde-DNP can be determined in the presence of a 100-fold excess of acetaldehyde-DNP. A 1000-fold excess of acetaldehyde-DNP does interfere. This is being checked over a range of formaldehyde-DNP concentration.

1-003E Instrumental Research - Mass Spectrometer (Varsel, Bell, Resnik, Harrow)

Fundamental studies on ionization potentials are being continued with the mass spectrometer. The ionization potentials are being calculated for known smoke constituents to determine the feasibility of low voltage mass spectroscopy for the quantitative analysis of smoke. To date the ionization potentials for nitrogen, argon, 1-butene, propene, methyl bromide, hydrogen cyanide, nitrous oxide, carbon dioxide, carbon monoxide, ammonia, n-butane, 1,3-butadiene, and methyl chloride have been calculated. The ionization potentials obtained by our techniques check the values reported in the literature within a 0.1 volt.

The smoke from regular treated Benson and Hedges cigarettes (WIN - tobacco extracted with heptane), from ventilated treated Benson and Hedges cigarettes (WIN-V - same as WIN only cigarettes were "ventilated"), and from regular untreated Benson & Hedges cigarettes was analyzed mass spectrometrically. The WIN showed only a slight isoprene reduction and the WIN-V a 25% reduction in isoprene when the spectra of the smoke from these cigarettes were compared to the spectra of the smoke from regular Benson & Hedges cigarettes. The 25% reduction in isoprene in WIN-V may very well be due to the dilution effect obtained with ventilated cigarettes.

The heptane content in the smoke from regular Benson & Hedges cigarettes was approximately 5 micrograms per cigarette. The heptane content was increased about 20 times in the WIN and 6 times in the WIN-V over that in the smoke from regular Benson & Hedges cigarettes.

1-003E-1 Instrumental Research - Electronics (Morrell, Harrow)

An ionization gauge, designed with parallel electrodes by Dr. Martin has been found to be superior to the concentric electrode type received from Aminco.

The effect of temperature variation of the cathode and anode were determined and the most beneficial effect of electrode heating was the elimination of noise caused by contamination on the electrodes.

A 0.3% mixture of natural gas in helium was prepared to obtain the sensitivity of this new gauge. The optimum gauge current value was determined by varying the power supply voltage while obtaining a signal from 1 ml. samples of the 0.3% mixture. This optimum current value at 4.5 mm was found

1001502385

<u>Chemical Tests</u>	
<u>Analyses</u>	<u>ATBL</u>
Total alkaloids	.85%
Petroleum ether extract	3.30
Ash	19.88
Total nitrogen	1.48
T.V.B.	.23
Sugar	5.5

ATBL from 6% NaOH Cooked Stem Pulp: ATBL films were made from 6% NaOH cooked stem pulp. Tobacco fines were added to the pulp in place of Molin's dust. The films were given to the Physical and Analytical Section for complete evaluation.

2-006B-2 Pilot Plant (Super, Long)

Two of our technicians have assisted Mr. Wilkinson of the Engineering Department in starting up the BL pilot plant. Our assistance consists of determining basis weights, binder weights, moistures, taking data, and observing the operating characteristics of the equipment.

2-006B-2 Physical Testing of BL Products (Stewart, Cosby)

The physical testing of two samples of ATBL from Mr. Butler has been completed. The physical testing of two samples of BL from the Richmond BL plant is in process.

2-006B-4 Sand Analysis on BL Materials (Pleasants, Wiley)

Analysis was completed on 253 samples.

2-006B-6 Binder Tag (Burnett)

13 samples have been prepared and forwarded to the National Spectrographic Laboratories, Inc. in Cleveland, Ohio for Spectrographic analysis of TiO_2 .

Filling Power (Burnett)

3 samples composed of 3 grades each of bright, burley and Maryland tobacco and 1 sample of the PM blend of Turkish tobacco have been collected and prepared for filling power and water and alcohol solubles content.

2-006B-11 BL Plant Assistance (Hind)

Experiments to date show that binders covered with CO_2 have a tendency to retain their viscosity while binders kept in air tend to lose viscosity when elevated to $50^\circ C$ temperature.

1001502392

A study of the smoking machines is being made in order to reduce the variance between the ports and between smokings, if possible.

/hj

cc: Dr. R. N. DuPuis
Dr. S. M. Cantor

Minutes of
SUPERVISORS' MEETING

August 29-30, 1957

Present: Dr. C. V. Mace
Dr. R. B. Seligman
Mr. L. L. Stewart, Jr.
Mr. S. W. Pleasants
Mr. J. Y. Mason, Jr.
Mr. A. P. Super
Mr. J. C. Holmes (Chairman)
Mr. P. E. Resnik
Dr. L. S. Harrow
Mr. J. T. Butler (Secretary)
Mr. W. H. Danker
Mr. G. H. Burnett

ADMINISTRATIVE

Dr. Mace commented that the summary of objectives, plans and accomplishments should be turned into the respective managers. He also requested that each supervisor prepare a list of the individual laboratory benches and office furniture within the next 10 days. In connection with plans for the new laboratory the following definitions will apply:

A writing table - a chemist's desk
Steam pressure definitions
low - 0 - 15 psi g
medium - 0 - 60 psi g
high - 60 and above psi g

TECHNICAL

1-001C Gas Chromatography (Murrill, Tignor, Gager)

Large quantities of CO₂ are removed from tobacco filler by sweeping the heated filler (100°C) with helium gas.

1-002F Polyhydric Alcohols (Martin, Carpenter)

The humectant present on the new Kent cigarettes has been identified as glycerol.

Known glycerol was quantitatively processed (100-101%) via the procedures developed in this laboratory. This included carbon column chromatography, reaction with phenylhydrazine, and mixed bed ion exchange chromatography.

Recoveries of known glycerol, after addition to uncased filler, ranged from 90 - 100% via the above procedures.

1001502384

2-methyl furan, have been obtained at room temperature. Plans are to investigate some compounds at elevated temperatures after which attempts will be made to monitor gas chromatographic columns.

-4-

Burning profiles of these cigarettes were determined and plotted. There does not appear to be any great difference between any of the three.

Efforts to puff the cellulose filler from Kent filter were continued. This has been a difficult problem because of the amount of dust found normally in the lab. However, recent tests with the cascade impactor and black millipore filters prove that some of the cellulose is dislodged on puffing under standard conditions for 16 hours.

1-010: Flavor and Odor Studies (Denker)

Employee Classification: The first series of tests were completed. 17 new good judges were discovered, 10 smokers and 7 non-smokers.

Herty-PM Blend Study: Preliminary tests show that 50% Herty can be detected by unlit draw and static smoke tests using difference from control method. A modified method will be used to evaluate lower Herty percentage.

Flavor Additives: A new meat smoke flavor added to plugs or filler shows promise. Most judges volunteered the comment that the flavored cigarette was milder. Most preferred the flavored cigarette over the control.

BL Program: About 3/4 of the production control samples were found to have the same quality aroma. The others have various different aromas.

Kent Cigarettes: The obviously stronger taste to unlit draw is being studied. A raisin type aroma is evident.

Dr. Seligman's group ran humectant studies.

Irritation Studies: A library search is being made of nose and mouth irritation physiology and irritation agents.

Smoke Ingredients: We are starting to make a collection and quantitative evaluation of smoke ingredients.

2-001 Aging of Tobacco (Crayton, Mason)

The individual effects of pressure, vacuum, and a methanol atmosphere during the forced aging method have been determined.

Pressure alone darkens the color of the leaf, though not uniformly, and has no effect on aroma.

Vacuum alone has no effect on leaf color but does give a milder aroma.

A methanol atmosphere has no effect on leaf color but does improve the aroma.

1001502387

-6-

2-003C Equilibrium Moisture Studies (Sharp, Mason)

The relationships between equilibrium moisture content and chemical composition have been determined for flue-cured, burley and Maryland tobaccos. The results indicate that at high humidities the E.M.C. is positively related to the water soluble constituents and negatively related to the cell wall materials. The reverse is true at low humidities. The humidity at which the relationships reverse appears to be 30% for flue-cured, 50% for burley, and 70% for Maryland.

2-005 Evaluation of Tobacco (Bass, Mason)

Burley Evaluation Test: Eight experimental lines and Kentucky 51A and Burley 21 were inspected on eight farmer plots in Kentucky.

Experimental lines 3 and 4 appeared to have the most desirable plant type, growth, and leaf quality in the field.

University of Kentucky personnel predicted the 1957 burley crop will be smaller in quantity and lower in quality than the 1956 crop. This is due to a less favorable growing season during 1957.

S. C. Stemmy Samples: Initial results from the 1957 S. C. tobacco indicate that the tobacco is of high quality.

P1 Strips, 1957 Crop, Greenville: A special sample of P1 strip was analyzed. The nicotine content and smoking characteristics of this tobacco were satisfactory for PM use.

Latakia Tobacco: A specially treated sample of Latakia tobacco was submitted by Mr. Kuhn.

The nicotine content of the test tobacco was similar to regular Latakia tobacco. The remainder of the analysis is incomplete.

Experimental Tobacco, Venezuela: Samples of burley and flue-cured tobacco grown on an experimental basis in Venezuela were analyzed. The effects of soil type, fertilization, irrigation, and varieties on leaf quality were determined.

The results on burley tobacco indicate that the frequency of irrigation may be reduced from every 6 days to every 10 - 12 days, and the addition of extra super phosphate is desirable.

DR 101 is a more desirable flue-cured variety than Coker 139 and DR 248. Irrigation every 10 - 12 days appears to be satisfactory, and the addition of extra fertilizer does not improve leaf quality.

1001502389

2-006B-11 Plant Assistance (Super, Long)

Tobacco Preparation: As reported last time, it has not been shown that any certain particle size fines adhere to the sheet preferentially, nor does sand adhere to the belt preferentially to dust.

The work has been completed except for some sand determinations which have not been done yet.

Desanding: The desanding section has been operated four times on both Class II and III by-products. The sand results have not been completed.

2-006B-12 Long Range Binder Studies (Hind)

All tobacco type sheets are easily removed when cast on Teflon sheets. To date, all sheets made on Teflon when removed showed no wet strength.

2-G14 Chico Cigarette Maker (Long, Waldron, Super)

The cutter sharpener has been altered to improve its performance.

A pressurized paste pot and paste assembly was borrowed from the Production Department and is being installed on the Chico.

A device for holding the top of the rod as it is cut is being considered as a method of improving the cutting.

A visit was made to the research laboratory of the American Tobacco Company to observe their Chico maker. The only major improvement that they had made on their machine was the installation of a separate drive on the feed hopper. The feed hopper drive was a Reeves adjustable drive with chain connections. Since this type of drive allows good control of the tobacco feed and cigarette weight, we may adopt it for our make.

Miscellaneous

Moisture Meter: A resistance type moisture meter for measuring the moisture content of sheet was loaned to us for testing by Minneapolis-Honeywell. It has not been possible to calibrate this meter because it will not give consistent reading on the same piece of BL. The meter takes about a minute to settle down and give a reading when a sample is measured.

Research Belt: A small BL processing belt is being designed for possible use by the Research and Development Department. Try out drawings are now being made.

1001502393

2-001 Aging of Tobacco (Crayton, Maron)

An oily aromatic substance was collected from flue-cured tobacco by pressure applied to alternate layers of tobacco and filter paper.

-5-

Pressure and vacuum darkens the color of the leaf uniformly but has no major effect on aroma.

Vacuum and methanol has no effect on leaf color but improves the aroma.

The results indicate that pressure, vacuum, and a methanol atmosphere are necessary to obtain maximum aging effects.

This work will be extended by sweating samples of the pretreated tobacco. Improvement in aroma should result from a sweat.

2-001B-1 Determination of the carbohydrates in Tobacco (Greene)

Variability in the reaction between oligosaccharides and anthrone must be eliminated before a carbohydrate comparison between tobacco types can be continued.

2-001B-2 Determination of the Alkaloids in Tobacco (Edmonds)

Paper chromatography revealed the presence of normicotine in highly concentrated samples of 99% pure nicotine. This impurity was removed by paper chromatography and the pure nicotine recovered. An 80% recovery of this nicotine was accomplished with the procedure developed in this laboratory. Efforts are being made to increase this recovery factor.

2-001C Petroleum Ether Extract - Chemical Studies (Brunot, Guvernator, Carpenter)

The hydroxamic acid test has been perfected so that 35 micrograms of glyceride can be detected.

A reverse-phase paper chromatographic system was used to separate mono-, di-, and tri-glycerides. Silicone fluid "200" rendered the paper hydrophobic, and chloroform-methanol-water (25:75:5) was used as the developing solvent.

The acids in the petroleum ether extract can be removed by ion exchange (or IRA 410, OH⁻ form), but the removal of the acids from the resin has not been accomplished.

Reverse-phase partition column chromatography is being evaluated for the separation of long chain aliphatic acids (C₁₅, C₁₈, etc.).

A sample of the total petroleum ether extract has been subjected to silica column chromatography. Hexane was used as the initial developing solvent, and the polarity of this solvent was gradually raised by additions of 5% ethyl ether. Pacificinic wax, squalene, some glycerides, and several unknown materials have emerged from the column and have been tentatively identified by instrumental means.

1001502388